AD A080145

(9) Research and development
~~R&D~~ Status Report,
~~Two Period~~
5 Sep[redacted]-5 Dec[redacted]979

(12)

(6) ADVANCED ALUMINUM ALLOYS
FROM
RAPIDLY SOLIDIFED POWDERS.

(14) LMSC-D683922

(11) Dec[redacted]979

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Sponsored by

Defense Advanced Research Projects Agency (DoD)

DARPA Order No. 3575

Monitored by
Air Force Materials Laboratory
Air Force Systems Command
United States Air Force
Wright-Patterson AFB, Ohio 45433

(15) Contract No. F33615-78-C-5203, DARPA Order-3575
Effective 5 September 1978 to 5 June 1982

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ADVANCED ALUMINUM ALLOYS FROM RAPIDLY SOLIDIFIED POWDERS

Advanced aluminum alloys are to be developed that will provide major payoffs for important new aircraft, spacecraft, and missile systems in the next decade. Payoffs will result from weight savings of structural components which, in turn, lead to increased range, payload, service life, and decreased life-cycle cost. Recently conducted feasibility and design tradeoff studies provide a basis for selecting certain property goals for improved aluminum alloys that will result in significant weight savings. These property goals are:

times 10 TO THE 6TH power

(A) Specific Elastic Modulus — 133 x 10^6 in.

(B) Specific Elastic Modulus — 122 x 10^6 in., and
Specific Yield Strength — 7.96 x 10^5 in. 10 TO THE 5TH power

Goal A is a 30-percent increase in specific modulus of elasticity relative to Al 7075-T76, without significant loss in strength, toughness, fatigue strength, or stress-corrosion resistance. Goal B is a 20-percent increase in specific modulus of elasticity accompanied by a 20-percent increase in specific strength, without significant loss in toughness, fatigue strength, or stress corrosion resistance.

1.0 OBJECTIVE

The objective of this program is to develop advanced aluminum alloys from rapidly solidified particulate that meet specific property goals. In addition, the program is to establish a metallurgical basis suitable for manufacturing scale-up and application to new weapon systems.

2.0 SCOPE

The program is divided into three phases, each consisting of a number of tasks. Phase 1 involves fundamental alloy development studies and consolidation process development and optimization. The most promising alloys are to be selected,

produced in simple mill form, and evaluated in Phase 2. Phase 3 will consist of a design evaluation using the properties of the alloys evaluated in Phase 2.

This program was initiated in September 1978 and is scheduled for completion in 3-1/2 years. The effort during the first two years will be devoted to Phase 1 only. This report describes activity during the reporting period in each of the four tasks comprising Phase 1.

3.0 PROGRESS

3.1 Task 1 - Development of Alloys Containing Lithium

This task is being conducted by the Lockheed Palo Alto Research Laboratories (LPARL).

3.1.1 Characterization of As-Extruded Alloys

Chemical analysis by atomic absorption has been performed on all eight first iteration extruded alloys made from un-screened splat particulate. The extrusion compositions are in reasonable agreement with the final melt compositions reported previously.

3.1.2 Heat Treatment of Extruded Alloys

Extrusions made from screened splat particulate (-8/+50 screen fraction) and extruded at 8:1 extrusion ratio, were solution treated at 811K (1000°F) for 0.5 h., water quenched, and aged at 473K (392°F) for 0.75, 8 and 100 hours, representing underaged, peak aged and overaged conditions. This screen fraction was selected in order to remove the majority of the coarse unsplatted particulate, and all of the fine, in-flight solidified particulate.

3.1.3 Characterization of Heat Treated Alloys

Tensile Properties. Tensile specimens were prepared from the three heat treatment conditions of alloys 1.2 (Al-3Li-2Cu-0.2Zr) and 1.7 (Al-3Li-0.5Fe-0.5Ni) and tested at room temperature. The results are shown in Table 1.

No improvement in elongation values was observed in these specimens, as compared to the recently reported results obtained on previous material made from un-screened particulate (Ref. 1). The yield strength values were found to be slightly lower than in the previously tested material, probably as a result of the slightly higher aging temperature used [473K (392°F) as compared to 464K (375°F) used previously].

It was also found that only small increases in elongation values were observed in the underaged and overaged conditions, as compared to the peak aged condition, for both alloys. Significant amounts of delamination were observed on the fracture surfaces, even in the specimens which failed by shear. The delaminations corresponded to failure along prior particle boundaries. It is suggested, therefore, that these prior particle boundary failures were the main cause of the relatively low elongation values observed, and the relatively small improvements in ductility in the underaged and overaged conditions. The use of higher extrusion ratios will therefore be investigated in the next stage of the work. A new extrusion die has been designed and is being made having an extrusion ratio of 20:1.

Microstructural Observations. TEM studies have been performed on consolidated and extruded material of alloy 1.7 (Al-3Li-0.5Fe-0.5Ni), in order to characterize the dispersoid particle and oxide particle distributions. Fine dispersoid particles were observed, in the size range 0.05 to 0.5 μm. Oxide particles were mostly found in bands corresponding to prior particle boundaries. Selected area diffraction patterns taken from the oxide particles indicated that the oxide is $LiAlO_2$ which exists in several polymorphic forms. The calculated d spacings agree most closely with the published values for the high pressure form of this oxide. Small amounts of porosity were also observed associated with the oxide particles. This porosity may be partly responsible for the observed tensile failures along prior particle boundaries.

3.2 Task 2 - Development of Non-Lithium-Containing Alloys

This task is being conducted by the Alcoa Laboratories.

3.2.1 Characterization of Loose Particulate

Surface analysis of fine atomized powder and splat particulate by SIMS/ISS* was completed. No inflight solidified particulate was examined; only true splats were selected for characterization. Table 2 shows that the splat oxide thickness is typically 30-40Å, while that of the powder is 50-80Å. Some selective diffusion of solute to splat surface was noted. The oxide on the splats exhibits a "low" degree of hydration.

3.2.2 Phase Stability Studies

Alloys 2.1A-2.4A, and 2.5A and 2.8A were selected for a study of the time-temperature dependency of structural decomposition and coarsening. The intent of the task is to identify a time-temperature range for hot pressing and extrusion which will yield a high volume fraction of precipitate phase but avoid serious coarsening. Loose flake material was aged 0.5, 5.0 and 50.0 hrs at 575, 675 and 775K (576, 756, and 936°F, respectively) and examined by TEM and x-ray analysis. The results show that all alloys exhibit little structural change at 575K and gross coarsening for extended (~5 hr) exposures at 775K. 675K appears to produce a good trade between precipitation and coarsening. x-ray results indicate the phase $MnAl_{12}$ precipitates in the Al-Mn alloys rather than $MnAl_6$.

The combination of hot press and extrusion temperatures (T_{HP}, T_{Ext}) of (775K, 575K); (675K, 675K); and (657K, 575K) has been selected for the first iteration fabrication.

3.2.3 First Iteration Alloys Fabrication

Problems were encountered in cold pressing the s 'at flakes by conventional wet-bag isostatic procedures. As a consequence, splat was cold compacted by a uniaxial mechanical die compaction process. Development of this technique resulted in some delays. After cold compaction, material was vacuum hot pressed according to usual practice. Distillation of relatively small amounts of butyl stearate, used sparingly as a die wall lubricant in the cold compaction, required slight modification of the vacuum degassing system to prevent

*Secondary Ion Mass Spectrometry/Ion Scattering Spectrometry.

contamination. Compacts were hot pressed October 15 - November 12 and then extruded the week of November 12-16, 1979.

The use of the three combinations of hot pressing and extrusion temperatures presented some problems in extrusion. These problems arise due to the anticipated high elevated temperature strength of these compositions. Utilizing the 2.27 Mkg (2500 ton) capacity press and a 24:1 extrusion ratio, either breakout could not be achieved, or extremely low ram velocities were obtained. According to a predetermined procedure, the extrusion (billet) temperature was raised in 28K (50°F) increments until breakout or significant ram velocity obtained. For all but two alloys intended to be extruded at 575K (576°F), extrusions were obtained utilizing a 602K (624°F) extrusion temperature. Alloys 513697 and 513693 (alloy 2.4A splat and powder, respectively), and alloy 513703 and 513658 (alloy 2.8A splat) required the use of 630K (675°F) or 700K (800°F) extrusion temperatures as indicated in Table 3. 700K (800°F) was selected since the billet failed to extrude at 630K (675°F), and it was desired to avoid excessive temperature increase over the hot pressing temperature of 675K (756°F). Excessive checking occurred in splat alloy 2.8A extruded at 630K (675°F) and powder alloy 2.8A extruded at 675K (756°F).

Mechanical property evaluation of these 35 extrusions has begun. Modulus determinations by ultrasonic pulse echo technique are being conducted by Drexel University.

3.3 Task 3 - Quantitative Microstructural Analysis and Mechanical Property Correlations

This task is being conducted by Georgia Institute of Technology. The following describe activity involving Al-Li alloy extrusions supplied by LPARL.

3.3.1 Tensile Properties

In addition to the tensile tests carried out on the underaged, peak aged and overaged conditions of alloys 1.2 (Al-3Li-2Cu-0.2Zr) and 1.6 (Al-3Li-1.5Mn), a few tests on alloy 1.2 were performed in the as-quenched condition. The

yield strength was found to be fairly low ($\sigma_{0.2} \approx 225$ MPa), and the elongation to fracture was not drastically higher ($\varepsilon_f \approx 8\%$) than the underaged condition of the same alloy ($\varepsilon_f \approx 2.8\%$).

The relatively low yield strength values of both alloys in the peak aged condition which are significantly below goal B, are also lower than the values reported previously (Table 9 of Ref. 1). One reason for this discrepancy may be the slightly higher aging temperature of 473K (392°F) used in the present case as compared to the aging temperature of 463K (374°F) for the previous specimens. A few tensile tests will therefore be performed on specimens aged at a temperature lower than 473K. The decreased aging temperature may increase the volume fraction of the δ' precipitates which should increase the yield strength.

3.3.2 Microstructural Observations

From TEM studies it was observed that the subgrain size in alloy 1.2 ranged from about 2 to 4 μm while in alloy 1.6 a size of about 5 μm was found. The δ' precipitates in the underaged condition exhibited sizes of about 100 to 200Å in diameter for both alloys. The precipitate free zones along grain boundaries are approximately 0.1 to 0.15 μm wide in both alloys.

The peak aged condition was studied so far only for alloy 1.6. The size of the δ' precipitates increased to approximately 200 to 300Å, as compared to the underaged condition. The width of the precipitate free zones also increased to about 0.2 μm.

A brief survey of one TEM - foil of alloy 1.6 in the overaged condition showed a somewhat surprising result: Superlattice reflections were not found in the diffraction pattern due to δ' - precipitates. Since it is known from previous studies that an aging treatment of 100 h. at 473K (392°F) does not lead to the dissolution of the δ' - precipitates, this preliminary result will be rechecked for other specimens.

3.3.3 Fracture Surface Observations

The low elongation to fracture values as well as the low true fracture strains, found for most of the aging treatments of alloy 1.2 and 1.6, may be due to delamination along the flake boundaries. Long cracks were observed on the surfaces of tensile specimens running parallel to the loading axis or extrusion direction. Cracks of this type were also found on the fracture surface of all tensile specimens. Crack nucleation seems to occur at or near the specimen surface for all aging treatments, even for the specimens of alloy 1.2 in the as-quenched condition. The nucleation of cracks at prior particle boundaries, decorated with oxide layers, would explain the minor variations of elongation to fracture values for the different heat treatments, although a large variation in strength was observed between the as-quenched, underaged, peak aged, and overaged conditions.

The fracture surfaces of the underaged and peak aged conditions of both alloys seem to indicate that, once cracks are initiated, the cracks propagate mainly along prior particulate, grain or subgrain boundaries. This type of fracture may lead to small dimples covering the individual grain or subgrain boundaries. The ductile rupture dimple spacing is approximately 0.5 μm. It is yet to be established whether these dimples are nucleated at grain boundary particles or formed by ductile tearing within soft precipitate free zones.

The fracture surfaces of tensile specimens in the overaged conditions of both alloys are somewhat different compared to the underaged and peak aged microstructures. The entire fracture surfaces as well as the specimen surfaces are covered with an unidentified layer, which could be the result of superficial surface corrosion to the laboratory air environment. It is therefore difficult to establish which feature on the fracture surfaces of the overaged conditions belong to the fracture process and which may be due to superficial corrosion.

It will be noted that similar surface effects were observed on polished samples prepared for light microscopy studi s. This corrosive effect seems to be more pronounced in alloy 1.6 than in alloy 1.2.

3.4 Task 4 - Application Studies

This task is being performed by Lockheed-California Company. There were no additional activities on this task in the report period.

4.0 MAJOR ITESM OF EXPERIMENTAL OR SPECIAL EQUIPMENT PURCHASED OR CONSTRUCTED DURING THE REPORTING PERIOD

None.

5.0 CHANGE IN KEY PERSONNEL DURING THE REPORTING PERIOD

None.

6.0 NOTEWORTHY TRIPS, MEETINGS, ETC. DURING THE REPORTING PERIOD

A contract review was held on September 12, 1979 at the Alcoa Laboratories, Alcoa Center, PA for AFML and DARPA. On September 13, 1979, an abbreviated review of the contract was presented to a number of invited guests from the Army, Navy, NASA, and various DoD aerospace contractors. Most of the guests gave brief reviews of contracts pertaining to development of advanced aluminum alloys using powder metallurgy techniques.

On September 17, 1979, H. G. Paris, F. R. Billman, and W. S. Cebulak, of the Alcoa Laboratories, presented a paper entitled "The Use of Rapidly Solidifed Al-Fe-Ni-Co, Al-Mn, and Al-Mn-Si Splat Alloys for Production of High Specific Modulus of Elasticity (E/ρ)", at the TMS-AIME Fall Annual Meeting, Milwaukee. At this same meeting,F. R. Billman, Alcoa Laboratories, presented a related paper entitled "Structural Characteristics of Splat Flakes Produced by the Single Drum Quenching Technique".

On October 23 and 24, 1979, a coordination meeting was held at Georgia Institute of Technology by Dr. I. G. Palmer of LMSC, Dr. H. G. Paris of Alcoa, Professors E. A. Starke, Jr., and E. E. Underwood, and Dr. T. H. Sanders of Georgia Tech to brief Dr. A. Gysler on contract activities to date. Dr. Gysler joined the Georgia Institute of Technology Fracture and Fatigue Laboratory Group on October 1, 1979, and will be studying microstructures in the Al-Li alloys. He was previously a principal scientist at DFVLR, the German Aerospace Research Establishment, Porz-Wahn, W. Germany. Prof. J. C. Williams and Dr. G. Lutjering of Carnegie-Mellon University were also in attendance. They will be performing some property-microstructure studies on two of the nonlithium-containing alloys.

7.0 SUMMARY OF PROBLEMS OR AREAS ON CONCERN IN WHICH GOVERNMENT ASSISTANCE OR GUIDANCE IS REQUIRED

None.

8.0 ANTICIPATED DEVIATION IN PLANNED EFFORT TO ACHIEVE CONTRACT OBJECTIVES

None.

REFERENCE

1. R. E. Lewis, "Development of Advanced Aluminum Alloys from Rapidly Solidified Powders for Aerospace Structural Applications", Interim Technical Report for Period March 1979 - September 1979, Air Force Contract F33615-78-C-5203, ARPA Order No. 3575, September 1979.

TABLE 1. TENSILE PROPERTIES OF CONSOLIDATED SCREENED Al-Li ALLOY PARTICULATE, SOLUTION TREATED AT 811K (1000°F) AND AGED AS INDICATED

Alloy	Nominal Composition	Extrusion #	Aging Time at 473K(392°F) (h)	0.2% Yield Strength (MPa)	0.2% Yield Strength (ksi)	Tensile Strength (MPa)	Tensile Strength (ksi)	Elongation (%)
1.2	Al-3Li-2Cu-0.2Zr	1.2A.1	0.75	413	60.8	491	71.2	4.7
			8	426	61.8	491	71.2	4.3
			100	285	41.4	373	54.1	5.2
1.7	Al-3Li-0.5Fe-0.5Ni	1.7A.1	0.75	274	39.7	355	51.5	3.8
			8	370*	53.7*	437*	63.4*	2.9*
			100	240	34.8	338	49.0	3.7

*Single specimen result; all others are average values of two tests

TABLE 2. **OXIDE THICKNESS OF NON-LITHIUM ALLOY SPLAT AND POWDER SAMPLES OBTAINED BY ION SCATTERING SPECTROMETRY**

Alloy	Nominal Composition	S-No.	Pot	Description	Oxide Thickness - Å Drum Side	Oxide Thickness - Å Atmosphere Side
Splat						
2.2A	Al + 2.3 Fe + 2.3 Ni + 4.6 Co	513687	2174	Argon Splat	26	24
2.2A	Al + 2.3 Fe + 2.3 Ni + 4.6 Co	513702	2189	Hot Air Splat	33	36
2.5A	Al + 9.7 Mn	513660	2148	Cold Air Splat	42	32
2.5A	Al + 9.7 Mn	513686	2175	Cold Argon Splat	20	26
2.5A	Al + 9.7 Mn	513704	2191	Hot Air Splat	135	131
2.5A	Al + 9.7 Mn + 2.5 Si	513689	2176	Argon Splat	51	32
2.6A	Al + 9.7 Mn + 2.5 Si	513705	2192	Hot Air Splat	43	35
2.8A	Al + 14.2 Mn	513658	2146	Cold Air Splat	33	26
Powder						
2.2A	2.3 Fe + 2.3 Ni + 4.6 Co	513682	--	--	55	--
2.6A	9.7 Mn + 2.5 Si	513666	--	--	80	--
2.8A	14.2 Mn	513678	--	--	50	--

TABLE 3. SCHEDULE OF FIRST ITERATION NON-LITHIUM ALLOY EXTRUSIONS+

S. No.	Sub#	T_{HP}	T_{EXT}	Sub#	T_{HP}*	T_{EXT}*	Sub#	T_{HP}*	T_{EXT}*	Particulate Form**	Alloy	Comment
513700	-1	935	625	-2	755	755	-3	755	625	S	2.1A	
513682	-1	"	"	-2	"	"				P	2.2A	
687				-1	"	"				ArS	2.2A	
702	-1	"	"	-2	"	"	-3*	"	"	S	2.2A	*513702-3A or 513702-3B
513701	-1	"	"	-2	"	"	-3	"	"	S	2.3A	
513697	-1	"	"	-3X	755	800	-3*	"	"	S	2.4A	*Only obtained 2.5 cm (1 in) pc
697	-1X	935	800							S	2.4A	
693				-1X	"	"				P	2.4A	
513704	-1	"	625	-2	"	755	-3*	"	"	S	2.5A	*513702-3A or 513702-3B
686				-1	"	"				ArS	2.5A	
513666	-1	"	"	-2	"	"	-3	"	"	P	2.6A	
705	-1	"	"	-2	"	"	-3	"	"	S	2.6A	
689	-1	"	"							ArS	2.6A	
513706	-1	"	"	-2	"	"	-3	"	"	S	2.7A	
513703	-1	"	675	-3X	"	800	-2	"	755	S	2.8A	
658							-1	"	675	S	2.8A	
678				-1	"	755				P	2.8A	Lot of edge cracks

+ 16.5 cm (6.5 in) dia. billet extruded to 1.3 X 7 cm (0.5 X 2.75 in) cross section

* T_{HP} = hot pressing temperature (K); T_{EXT} = extrusion temperature (K)

**S = splat particulate, P = fine atomized powder, ArS = splat particulate produced in argon gas environment.